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For: AN EXTRUDABLE THERMOPLASTIC MATERIAL AND A FIBER MICROMODULE MADE FROM SUCH A MATERIAL

DECLARATION

I, Andrew Scott Marland, of 35, avenue Chevreul, 92270 BOIS COLOMBES, France, declare that I am well acquainted with the English and French languages and that the attached translation of the French language PCT international application, Serial No. **PCT/FR00/02545** is a true and faithful translation of that document as filed.

All statements made herein are to my own knowledge true, and all statements made on information and belief are believed to be true; and further, these statements are made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any document or any registration resulting therefrom.

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AN EXTRUDABLE THERMOPLASTIC MATERIAL AND A FIBER MICROMODULE MADE FROM SUCH A MATERIAL

The present invention relates to an extrudable material suitable for making thin films, the material including an olefin polymer. A major, although non-exclusive, application of the invention lies in making the sheaths of sheathed optical fiber micromodules suitable for incorporation in a cable such as the cable described in document EP—A—0 468 878, to which reference can be made.

In certain applications, and in particular when making micromodules comprising a bundle of sheathed optical fibers in mutual contact enclosed together with a sealing gel inside an extruded support covering, it is desirable to satisfy conditions which are contradictory to some extent. For example, particularly when making micromodules, it is often desirable to achieve the following simultaneously:

· suitability for extrusion as a film that is thin (if possible about 0.1 mm thick);

· compatibility between the material and the usual sealing gels;

· sufficient strength for the material once in the form of a thin film to make handling possible during subsequent operations without any risk of tearing;

· no adhesion between the film of the micromodule sheath and the fibers during the heating that occurs when the outer covering of thermoplastic material is put into place;

· a proper cylindrical shape is maintained during manufacture of the micromodule and while micromodules are being assembled together to make up a cable;

· little shrinkage during extrusion of the sheath to make the micromodule or during cooling, so as to avoid stressing the optical fibers;

· the material should be easy to color so as to enable the micromodules to be identified;

· the material should be stretchable to a limited extent only so as to make it easy to strip a micromodule in order to prepare its ends for fiber connection; and

· a high degree of resistance to the chemicals used during the operations that are preformed on cables, for example resistance to cleaning solvents.

When manufacturing optical fiber cables, some of the above characteristics are essential, in particular mechanical strength, including during thermal aging, and compatibility with sealing gels and cleaning solvents used for removing gel and dirt prior to connecting the optical fibers to a connector. However mechanical strength is unfavorable to ease of use since a strong sheathing film that presents a high level of breaking elongation makes it difficult to strip micromodules in order to release the end portions of the fibers.

An electrically insulating material is already known (GB—A—2 110 696) that comprises an alloy of polymers that are crosslinked at least in part, containing in particular a copolymer of ethyl alkyl acetate (EVA) having more than 40% vinyl acetate with inorganic fillers at a concentration sufficient to make the material fireproof. Crosslinking is for the purpose of making it possible to have a high concentration of fillers.

The present invention seeks in particular to provide a material that can be extruded as a thin film and that presents a satisfactory compromise between the various results to be achieved. For this purpose, the invention proposes in particular a material that can be extruded as a thin film, the material being constituted by a composition containing at least one (and possibly a plurality of) practically non-crosslinked thermoplastic olefin polymer and a filler content lying in the range 25% to 65% by weight of the composition, said material in the non-divided state having traction strength lying in the range 6 megapascals (MPa) to 20 MPa and breaking elongation lying in the range 50% to 300%.

The term "practically non-crosslinked" is used to mean a polymer that is described in that way in the trade, and consequently having no appreciable degree of crosslinking and containing no crosslinking agents such as peroxides, other than in trace amounts.

Because of the absence of any crosslinking, the presence of "gels" is avoided, where such gels impede thin-film extrusion, and post-extrusion shrinkage which would give rise to stress on the fibers is reduced.

The hardness of the material on the Shore D scale advantageously lies in the range 35 to 55.

When using the material to make a micromodule sheath, selecting hardness on the Shore D scale that exceeds 35 makes it possible to guarantee a satisfactory cylindrical shape and to avoid the "kinking" effect whereby a sharp bend or "kink" can form during the bending that is necessary for making connections.

Because of the limited breaking elongation, due in particular to the presence of the filler, stripability is satisfactory and there is no need to have recourse to special tools. The above minimum characteristics, and in particular traction strength and breaking elongation ensure that the material is not excessively fragile during handling. In particular, the above minima make it possible to perform the handling required for cable manufacture or for making connections without excessive risk of damage.

The above-mentioned minimum filler concentration serves to reduce expansion and contraction of the materials during the variations in temperature that occur during cable manufacture. The presence of a sufficient quantity of fillers makes it possible to avoid any risk of the micromodules sticking to one another, to the sheathed fibers, or to an outer covering.

The fillers used are generally mineral fillers. In particular, it is possible to use (optionally hydrated) alumina, chalk, kaolin, talc, silica, magnesium

hydroxide, and mixtures thereof. All of these fillers reduce breaking elongation and expansion or contraction during temperature variations. Furthermore, they increase thermal inertia and heat capacity. The maximum filler concentration mentioned above enables viscosity to be maintained at a level which is compatible with extruding a thin film.

Suitable olefin polymers are substantially the same as those commonly used at present. In particular, mention can be made of the following substances:

· PE: polyethylenes;

· PP: polypropylenes;

· EPR: ethylene propylene rubber;

· EPDM: ethylene propylene diene monomer;

· EVA: copolymers of ethylene and lower alkyl acetates (in particular vinyl acetate);

· EBA: copolymers of ethylene and lower alkyl acrylates;

· EEA: ethylene ethyl acrylate;

· EMA: ethylene methyl acrylate;

· VLDPE: very low density polyethylene;

· acrylic acid or maleic anhydride grafted polymers;

· PVC: polyvinyl chloride;

· mixtures and copolymers thereof.

The various polymers are not fully equivalent to one another. Often a mixture of olefin polymers should be used where one of the components is PE or PP and the other is selected from the other polymers mentioned above.

If the second polymer is EVA, then a compound should be used having no more than 30% of the vinyl acetate comonomer in order to conserve sufficient hardness and mechanical characteristics. EBA, EEA, and EMA have properties similar to EVA. EPR and EPDM should be used with concentrations of ethylene that are high enough to prevent them having properties that come close to those of an elastomer.

When using a polymer made up firstly of PE or PP and secondly of EVA copolymer, it is advantageous to use a composition having 40% to 80% EVA.

In general, the extrudable material should also include a low concentration (not exceeding a few percent by weight) of plasticizing agents such as aliphatic oils or phthalates (e.g. dioctyl or didecyl phthalate), adipates, trimellitates, etc.

Substances for providing protection against heat or ultraviolet radiation are incorporated when exposure to sunlight is to be expected.

In some cases, one or more silanes or aminosilanes should be added, such as:

. vinyl trimethoxysilane;

. amino propylsilane;

. amino trimethoxysilane.

If trialkoxy silane is used, it is desirable to avoid using compounds having more than five carbon atoms.

Silanes serve to reinforce bonding between the filler and the polymer.

In the absence of a crosslinking agent, silane does not run any risk of giving rise to crosslinking which in addition would not be possible when using the material for making optical fiber sheaths, since the temperatures required for crosslinking are then not reached during extrusion.

The invention also provides an optical fiber micromodule comprising a bundle of optical fibers and a sheath surrounding the bundle that is made of a thin film of an extrudable material, the micromodule being characterized in that the sheath is constituted by a composition containing a thermoplastic olefin polymer and a filler content lying in the range 25% to 65% by weight of the composition, said material in the non-divided state having traction strength lying in the range 6 MPa to 20 MPa and breaking elongation lying in the range 50% to 300%.

The properties of several materials in accordance with the invention are given below by way of example together with a comparison with a reference material conventionally used at present for making micromodule sheaths.

The description refers to the sole figure which shows a micromodule in a deformed state that it is likely to take up when pressed against other micromodules by an outer covering.

The micromodule comprises a plurality of individually-sheathed optical fibers 10 contained in a sheath 12 which needs to be easily torn so as to enable the ends of the fibers to be stripped for connection purposes. The sheath 12 is generally constituted by being extruded onto the bundle of optical fibers 10 while they are being drawn down, and it then takes on a shape that is approximately circular providing the outline presented by the bundle of fibers is itself not too far removed from the circumscribing circle. The sheath presses tightly against the fibers and causes them to press against one another. Within a cable, the pressure exerted by micromodules on one another can deform the sections of the micromodules and can cause them to take up the shape as shown, for example.

The reference material was constituted by polyethylene having nominal specific gravity of 0.92 and a melt flow index of 0.3 grams per 10 minutes (g/10 min) at 190°C under a pressure of 21.6 newtons (N). That material was used to make a micromodule sheath by being extruded onto a bundle of four optical fibers. The sheath 12 made in that way had a diameter of 1 millimeter (mm) and a thickness of 0.12 mm. Extrusion took place without difficultly and the resulting sheath was indeed cylindrical. However, while the cable was being made up, by extruding an outer covering based on polyethylene, the heat required for extruding the covering deformed the micromodules and their sheaths tended to stick to one

another and to the outer covering, thus requiring special precautions to be taken such as interposing one or more separator tapes between the micromodules and the covering.

Those difficulties are avoided when using a material in accordance with the invention.

Example 1

A mixer was used to prepare a composition comprising the following by weight:

· 50 parts of polyethylene having specific gravity of 0.92 and a melt flow index at 190° under 21.6 N of 1.8 g/10 min;

· 50 parts EVA copolymer containing 18% vinyl acetate;

· 130 parts alumina hydrate;

· 5 parts lubricant (paraffin oil); and

· 5 parts additives (antioxidizers, silane, lubricant).

The ingredients were mixed for 10 minutes up to 160°C.

After calendaring on a cylinder mixer, the material was cut up and then molded under pressure at 180°C into the form of plates suitable for performing measurements for characterizing the material.

The mechanical characteristics obtained on the plates were as follows:

· breaking strength = 11.4 MPa;

· breaking elongation = 125%;

· hardness = 45 on the Shore D scale.

The composition was used for making micromodules. For that purpose, it was converted into granules which were inserted into an extruder having a diameter of 45 mm, and a length of 24 diameters.

Extrusion temperatures lay in the range 130°C to 165°C going from the feed hopper to the extrusion head.

In order to characterize the resulting sheath, two operations were performed.

The first was forming at a speed of 100 meters per minute (m/min) in order to obtain a tube having an outside diameter of 0.90 mm and a radial thickness of 0.12 mm.

For the second operation, the forming was identical to the above except that four colored optical fibers were introduced through the extruder head and a sealing gel was injected simultaneously so as to form a module which, once the extruded material had cooled, was collected in a bin where it was allowed to coil freely while flat.

The characteristics obtained on the sheaths were as follows:

	Module without sealing gel	Module with gel
Initial characteristics	BS = 4.5N BE = 138%	BS = 4.6N BE = 112%
Winding on a 6D mandrel	OK	OK
After 10 days at 70°C	δBS = 19% δBE = 15%	δBS = 13% δBE = 13%
After 10 days at 70°C + 42 days at 80°C	δBS = 17% δBE = 20%	δBS = 9 δBE = 11%

BS: breaking strength expressed in Newtons

BE: breaking elongation, expressed in %

δ: change

These results show that sheaths made of the material of the invention present firstly good resistance to heating, and secondly good compatibility with the filler materials.

Example 2

The composition of the material was identical to that of Example 1, except that the filler based on hydrated alumina was replaced by a filler based on calcium carbonate. Mixing was performed under the same conditions, and a micromodule having a diameter of 0.8 mm and a thickness of 0.11 mm was extruded at 100 m/min. The characteristics below show how such a formulation provides modules having adequate chemical resistance in spite of the small thickness of the module sheath.

Initial characteristics	BS = 3.9N	BE = 155%
After 1 hour in ethanol at 20°C	δBS = 1%	δBE = 3%
After 1 hour in isopropanol at 20°C	δBS = 5%	δBE = 3%

Example 3

A formulation identical to Example 1 was made except that the alumina-based filler was replaced by a kaolin filler, and its concentration was reduced to 65 parts. The paraffin plasticizer was replaced by an oil of the isononyl adipate type.

The various ingredients were introduced into an internal mixer, mixed at up to about 160°C, and granulated. The characteristics of the material in plate form were as follows:

Initial mechanical characteristics	BS = 10.5 MPa	BE = 157%
10 days aging at 70°C	δBS = +1%	δBE = -13%
42 days of aging at 80°C	δBS = +7%	δBE = -19%
Compatibility with Macroplast CF 300 jelly 10 days at 70°C	δBS = -15%	δBE = -18% Change in mass = 7%
Ability to withstand humidity at high temperature, 42 days at 40°C and 93% relative humidity	δBS = -4%	δBE = -+2%
24 hours immersion in lamp oil at 20°C	δBS = -25%	δBE = -10%
1 hour immersion in ethanol at 20°C	δBS = -4%	δBE = -10%
1 hour immersion in isopropanol at 20°C	δBS = -6%	δBE = -4%
1 hour immersion in isopropanol at 20°C	δBS = -4%	δBE = -10%
Hardness	45 on the Shore D scale	

Using the same formulation, a micromodule having four optical fibers was made under the same conditions as before to have a 0.11 mm thick sheath with a diameter of 0.85 mm. The sealing gel was "Macroplast CF 300" from Henkel.

The mechanical characteristics (mech. ch.) obtained on the module were as follows:

Initial mech. ch.	BS = 2.4N	BE = 105%
Changes in mech. ch. after 10 days at 70°C	δBS = 5%	δBE = 4%
Changes in mech. ch. after 10 days at 70°C in Macroplast CF 300	δBS = 0	δBE = 6%
Changes in mech. ch. after 42 days at 80°C	δBS = 2%	δBE = 5%
Changes in mech. ch. after 10 days at 70°C in CF 300, and 42 days at 80°C	δBS = -21%	δBE = -6%
Changes in mech. ch. after 42 days at 40°C and 93% relative humidity	δBS = 5.4%	δBE = 0%
Changes in mech. ch. after 24h in lamp oil	δBS = 11%	δBE = 25%
Changes after 24 hours in ethanol	δBS = 8%	δBE = 12%
Changes after 24 hours in isopropanol	δBS = 4%	δBE = 13%